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The effect of various growth parameters such as temperature, V/III ratio and the growth rate on the properties of InN layers grown by MOCVD was investigated. The InN layers were deposited onto 2 μm thick GaN-on-c-plane sapphire films. In addition, the different precursor injection procedures were investigated. Since the growth of InN required very low deposition temperatures around 600 °C, for the deposition of InN/GaN heterostructures similar experiments were performed to optimize the growth of GaN at comparable growth temperatures.

The fabrication of GaN/InN/GaN structures for device applications was complicated by intermixing and surface segregation of indium and defect formation in heterostructures related to the large lattice mismatch of 10% between GaN and InN.

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Fabrication of AlGa_N-Ga_N-In_N high electron mobility transistors

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Abstract :

The effect of various growth parameters such as temperature, V/III ratio and the growth rate on the properties of In_N layers grown by MOCVD was investigated. The In_N layers were deposited onto 2 μm thick GaN-on-c-plane sapphire films. In addition, the different precursor injection procedures were investigated. Since the growth of In_N required very low deposition temperatures around 600 °C, for the deposition of In_N/GaN heterostructures similar experiments were performed to optimize the growth of GaN at comparable growth temperatures.

The fabrication of GaN/In_N/GaN structures for device applications was complicated by intermixing and surface segregation of indium and defect formation in heterostructures related to the large lattice mismatch of 10% between GaN and In_N.

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The fabrication of GaN/InN/GaN(AlGaN) heterojunctions required firstly the development of an InN growth process in our reactor, secondly the optimization of GaN growth at temperatures compatible to InN, and finally the investigation of GaN/InN/GaN heterostructures. The growth of GaN/InN/GaN structures is extremely challenging due to the large lattice mismatch of 10% between GaN and InN, and the low thermal stability of InN.

All layers were grown by MOCVD on 2 μm thick semi-insulating GaN on c-plane sapphire substrates¹ using the precursors trimethylindium (TMIn), trimethylgallium (TMGa), triethylgallium (TEGa) and ammonia. The InN layers were deposited at temperatures between 400 and 700 °C. Thereby, the TMIn and ammonia flows were varied between 0.6 – 33 $\mu\text{mol/min}$ and 0.08 – 0.33 mol/min, respectively. The carrier gas was nitrogen. The total gas flow and the reactor pressure were kept constant at 12 l/min and 760 Torr, respectively.

1. Growth of InN bulk layers in the conventional growth mode

To investigate the effect of the growth temperature on the growth behavior, about 200 nm thick InN layers were deposited at temperatures between 400 and 700 °C on (111)Si substrates using a TMIn flow of 33 $\mu\text{mol/min}$. The layer thickness was determined by ellipsometry. The growth rate of InN, r_{InN} , increased with increasing temperature in the range 400 °C < T_{gr} < 600 °C. However, at $T_{\text{gr}} > 620$ °C, r_{InN} drastically decreased with increasing T_{gr} due to the instability of InN at higher temperatures, and In droplet formation dominated (Fig. 1).

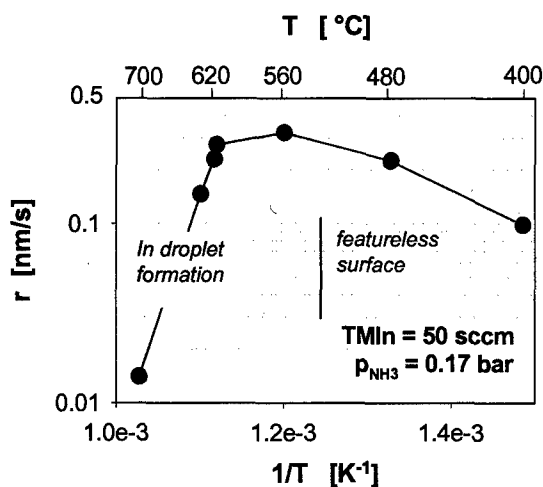
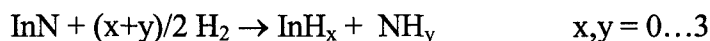


Fig. 1. Dependence of the growth rate on the growth temperature for InN film grown in the conventional growth mode.

The InN growth rate was also strongly affected by the NH₃ flow in the reactor (Fig. 2). At a deposition temperature of 480 °C, the growth rate followed the expected trend: r_{InN} increased with rising NH₃ injection and saturated at higher NH₃ flows. In contrast, at a deposition temperature of 620 °C, a drop in growth rate at higher NH₃ flows was observed. We believe that this growth rate reduction is a result of an increased hydrogen partial pressure in the reactor, originating from the decomposition of NH₃.



Using the same growth conditions as for the growth rate calibration, InN layers were also deposited on thick GaN base layers. When characterized by X-ray diffraction, a broadening of the FWHM of the (002) reflection from 580 to ~1000 arcsec was observed with increasing growth temperature. Note, that all InN layers, which were deposited under these non-optimized conditions, grew in a 3 dimensional growth mode as illustrated in Fig. 3b. The increase in the InN FWHM just reflected the more random grain orientation at the higher InN deposition temperatures.

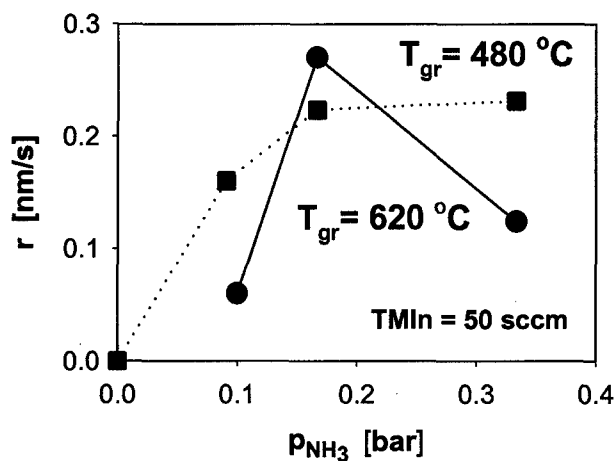


Fig.2. Dependence of the InN growth rate on the NH₃ partial pressure in the growth chamber

Hall measurements performed on the same samples showed a steady decrease in the carrier concentration with rising deposition temperature from $2.8 \times 10^{20} \text{ cm}^{-3}$ to $3 \times 10^{18} \text{ cm}^{-3}$ for films grown at 400 and 620 °C, respectively. The observed temperature trend is most likely related to a reduction in the residual oxygen impurity incorporation at higher deposition temperatures, as the formation of In oxides becomes less favorable.

Since the efforts under this program were directed towards the fabrication of heterostructures with very thin InN layers, we continued our investigations searching for conditions, which would allow the InN layer to grow in a step flow growth mode similar to GaN. If not mentioned otherwise, all following experiments were performed at a growth temperature of 600 °C, to minimize impurity incorporation into the layers and also to promote surface diffusion of adsorbed species. To counteract the generally low surface mobility of species at these low growth temperatures, we drastically reduced the growth rate of the InN layers to values as low as 0.005 nm/s. However, despite of the extremely low growth rates the InN films still grew in a 3 dimensional growth mode as seen in Fig. 3b. For this reason we decided to investigate pulsed and alternate precursor injection schemes which are well known to enable step flow growth of conventional III-V and II-IV semiconductors at low growth temperatures.

2. InN growth using modulated precursor injection schemes

Two different injection schemes were investigated a) atomic layer epitaxy (ALE) where TMIn and NH_3 were alternately injected into the growth chamber (Fig. 4a) and b) pulsed injection of TMIn while NH_3 was continuously supplied (Fig. 4b). When TMIn and NH_3 were injected alternately (ALE growth mode), InN island arrays formed on the terraces of the underlying GaN layer (Fig. 3a). Each island consisted of small steps and terraces (Fig. 3c). Grain formation could also be suppressed using pulsed TMIn injection as seen in the AFM Fig. 3d. Although a tendency towards island formation is visible in this image too, the islands appear flatter compared to the sample grown in the ALE growth mode.

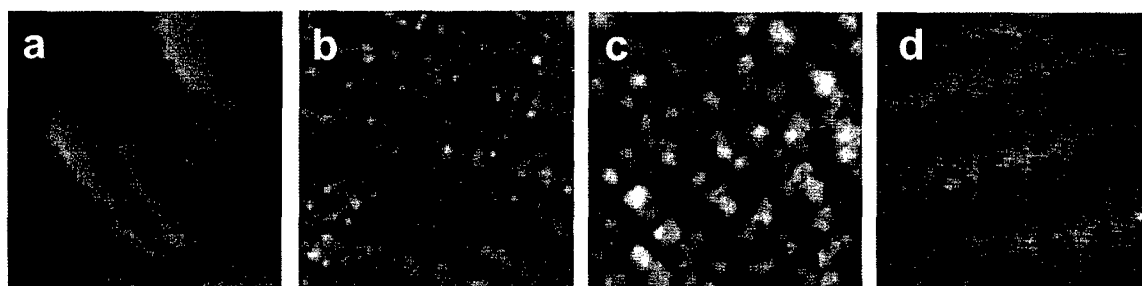


Fig. 3. Atomic force microscopy images of (a) the 2 μm thick GaN base layer, (b) 2 nm InN grown in the conventional growth mode, (c) 2 nm InN grown in a pulsed growth mode and (d) 2 nm InN grown by ALE (image size 500 x 500nm, grayscale 3 nm).

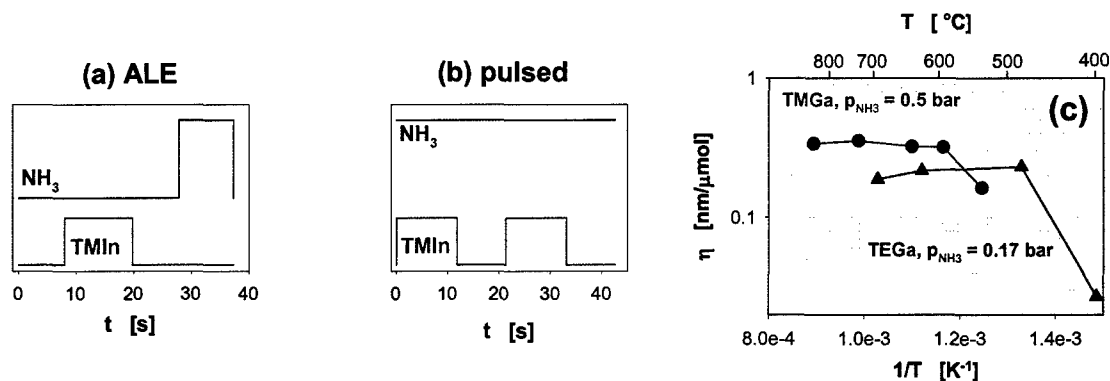


Fig. 4. Precursor injection schemes used for the deposition of thin InN layers grown (a) in the pulsed and (b) in the ALE growth mode; (c) temperature dependence of the GaN growth efficiency using TMGa and TEGa as precursor for conventional growth.

The InN island formation is most likely related to the large lattice mismatch between the GaN and InN layers (10%). Whereas the ALE growth procedure (4a) drastically suppressed the formation of metal droplets on the surface, droplets were still found on the surfaces of films grown using pulsed TMIn injection, due to the simultaneous presence of TMIn and NH_3 in the gas phase. Note, that the overall growth rate of the InN films deposited using procedures (4a) and (4b) was lower than extrapolated from the thickness of layers deposited in the conventional growth mode, possibly related to desorption of species from the surface in the “zero-growth” phase of the injection cycle.

The electrical characterization of 1 – 2 nm thick InN films grown on S.I. GaN base layers indicated that the carrier concentration in the layers increased with decreasing growth rate and flush/growth interruption time in the cycle. Thus, for the thin InN layers grown using procedure (4a) and (4b) free carrier concentrations in the order of 10^{20} cm^{-3} were measured. The exact determination of the carrier and impurity concentrations, however, was complicated by the extremely low overall growth rates and the resulting difficulties in the exact determination of the layer thickness. The high residual carrier concentration could be related to an enhanced formation of nitrogen vacancies. However, a more likely explanation is, that the alternate growth modes lead to a reduction in the residual carbon concentration and thus a reduced compensation of the InN films, while the oxygen impurity incorporation is unaffected, resulting in an overall higher free carrier concentration.

3. Low temperature growth of GaN

The fabrication of InN/GaN heterostructures required the development of a GaN growth process compatible to that for InN. To explore the growth of GaN at low temperatures (LT), TEGa was also investigated as an alternative precursor to TMGa. Using TEGa instead of TMGa, the transition temperature between diffusion and kinetically controlled growth was reduced from 580 to 480 °C (Fig. 4c). However, similar to InN, 3 dimensional growth of GaN was observed at $400^\circ\text{C} < T < 620^\circ\text{C}$ for conventionally grown films, even at growth rates as low as 0.005 nm/s, independent of the Ga precursor (Fig. 5a and 5d). At $T = 620^\circ\text{C}$ and injecting TEGa and NH_3

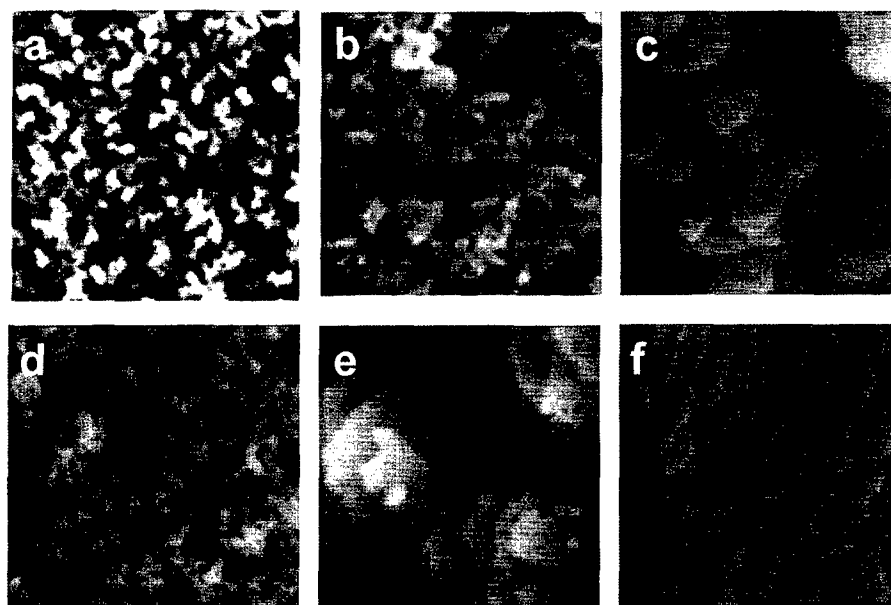


Fig 5. AFM images of 3nm thick GaN layers deposited at 620 °C on 2 thick GaN base layers using TEGa as precursor and (a) the conventional growth mode, (b) ALE, (c) pulsed TEGa injection; and using TMGa as precursor and (d) the conventional growth mode, (e) ALE, and (f) pulsed TMGa injection, (image size 500 x 500 nm, greyscale 3 nm).

alternately (ALE), grain formation could be suppressed, but no smooth steps were formed (Fig. 5b). However, step flow growth occurred using pulsed injection of TEGa (Fig. 5c). Surprisingly, the most regular step structure of the surface was obtained for pulsed injection of TMGa (Fig. 5f). The injection sequence was 24 s TMGa, 24 s interruption (Fig. 2c). SIMS investigation of GaN films grown at a temperature of 620 °C using conventional carrier injection as well as pulsed TMGa-precursor injection (see Fig. 6) revealed a significant reduction of residual carbon and oxygen contaminants in the layers grown in the pulsed growth mode ($[O] \sim 2 \times 10^{19} \text{ cm}^{-3}$, $[C] < 1 \times 10^{18} \text{ cm}^{-3}$), compared to the conventionally grown layers ($[O] \sim 2 \times 10^{20} \text{ cm}^{-3}$, $[C] \sim 5 \times 10^{19} \text{ cm}^{-3}$). As expected the C impurity concentration in layers grown with TEGa was somewhat lower ($[C] \sim 8 \times 10^{18} \text{ cm}^{-3}$). The oxygen concentration determined in the SIMS measurements corresponded closely to the free carrier concentration determined for thin GaN films deposited on S.I. GaN base layers, which was in the order of $(2 - 5) \times 10^{19} \text{ cm}^{-3}$.

4. GaN/InN/GaN heterostructures

Depositing the InN layer by ALE (Fig. 4a) and the low temperature GaN layer in the pulsed growth mode ($T_{\text{gr}} = 620 \text{ °C}$), 3 nm GaN/ (0.5 –2) nm InN/ 2 μm S.I. GaN DHs with smooth surfaces were obtained, as seen in the AFM image displayed in Fig. 7a.

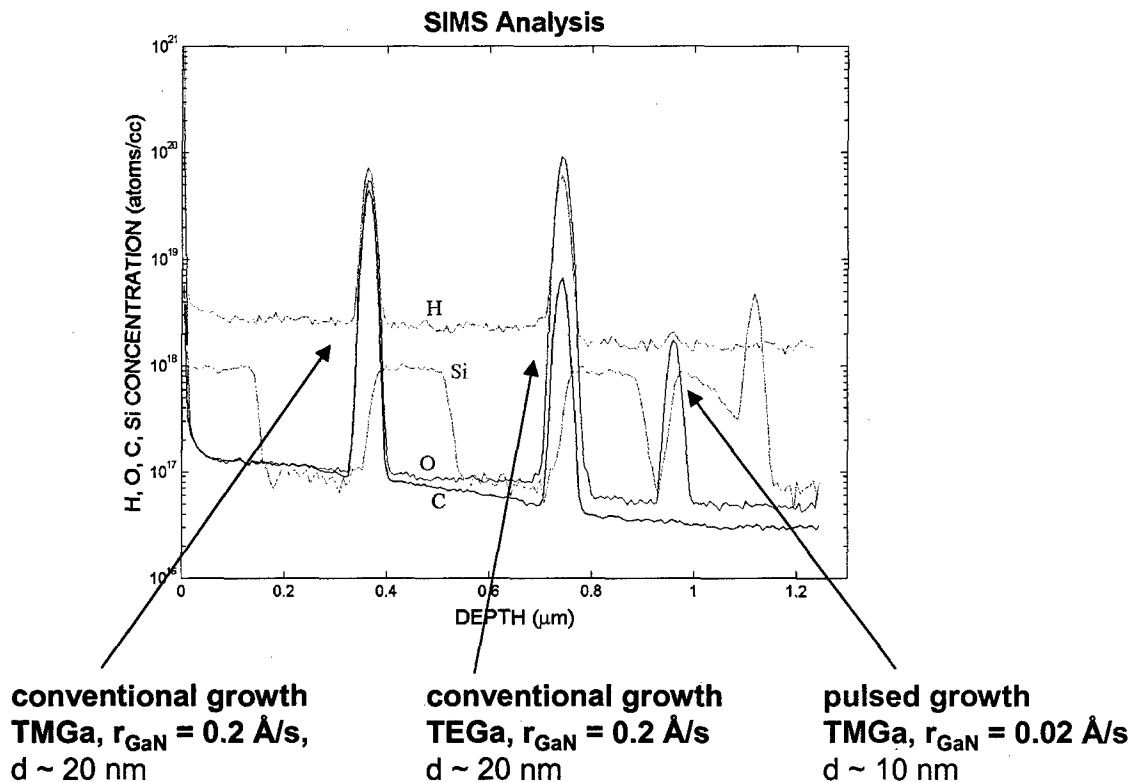


Fig. 6. SIMS analysis of thin GaN layers deposited at 620 °C, which were embedded in GaN grown at high temperature.

However, the AFM images also indicates, that new structural defects were formed during deposition of the GaN cap layer. Similar defect formation was observed when the 2 nm thick InN layer was deposited in the pulsed growth mode (Fig. 7b). Possibly, the InN islands grown on GaN are partially relaxed, resulting in defect formation, in particular during the deposition of the GaN cap layer due to the large lattice mismatch between InN and GaN ($\sim 10\%$). Similar observations had been made for InN quantum dots deposited by MBE.² Hall measurements revealed extremely high sheet carrier densities (n_s) for these heterostructures. For an InN thickness of about 0.5 and 10 nm, n_s values of 2.6 and $3.3 \times 10^{13} \text{ cm}^{-2}$ were measured. Taking into account that the cap layer alone contributed a charge of $n_s \sim 1.5 \times 10^{13} \text{ cm}^{-2}$, the free carrier concentration of the InN layers was in the order of 10^{20} cm^{-3} , as discussed in the previous paragraph. The corresponding electron mobility values varied between 180 and 220 cm^2/Vs . However, we believe, that due to the extremely high carrier concentration in combination with the low thickness of the channel, the carriers were not confined in the channel and spilled over into the GaN layer. The measured electron mobility should then correspond to the conduction of electrons in GaN. Also, PL measurements of the same samples did not show any InN related luminescence. Instead we observed a luminescence peak at a wavelength of 390 and 400 nm, for an estimated InN layer thickness of 0.5 and 1 nm, respectively (Fig. 3). Obviously the InN layer intermixed with the GaN during GaN cap layer growth. The generally lower In content in comparison to observations made during MBE growth (88 % In at $T_{\text{gr}} = 400 \text{ }^\circ\text{C}$) may be related

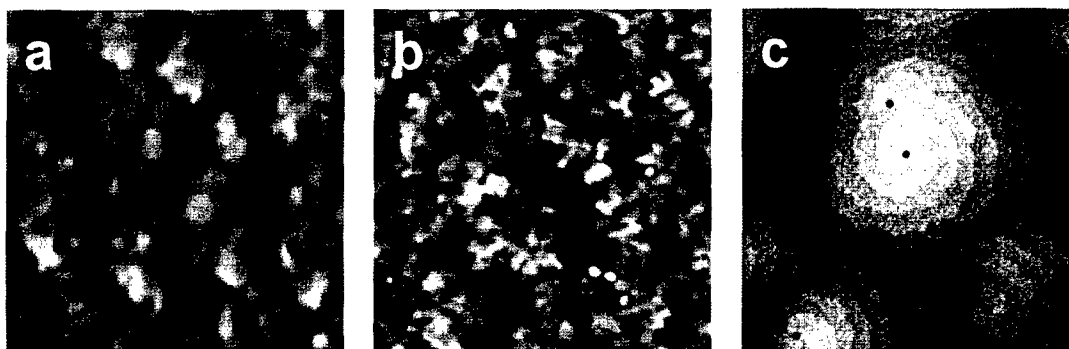


Fig. 7. AFM images of GaN/InN/GaN samples with (a) 2nm InN grown by ALE/ 3 nm GaN cap grown in the pulsed mode, (b) 2nm InN / 3 nm GaN cap both grown in the pulsed mode, and (c) a GaN/10 nm $\text{In}_{0.15}\text{Ga}_{0.85}\text{N}$ / 2 nm InN structure. The InN layer was grown in the pulsed mode (image size 500 x 500 nm, grayscale 3 nm).

to the higher solubility of GaN in InN and vice versa at the significantly higher growth temperature of the layers in the present study ($T_{\text{gr}} = 620\text{ }^{\circ}\text{C}$).³ The high residual oxygen impurity concentration in the films obviously did not perturb the luminescence too much, as oxygen acts as a shallow donor in group-III nitrides and behaves similar to silicon.⁴ Note that intermixing and In segregation was also observed during attempts to grow InN/ GaN multilayerstructures for SIMS investigations.

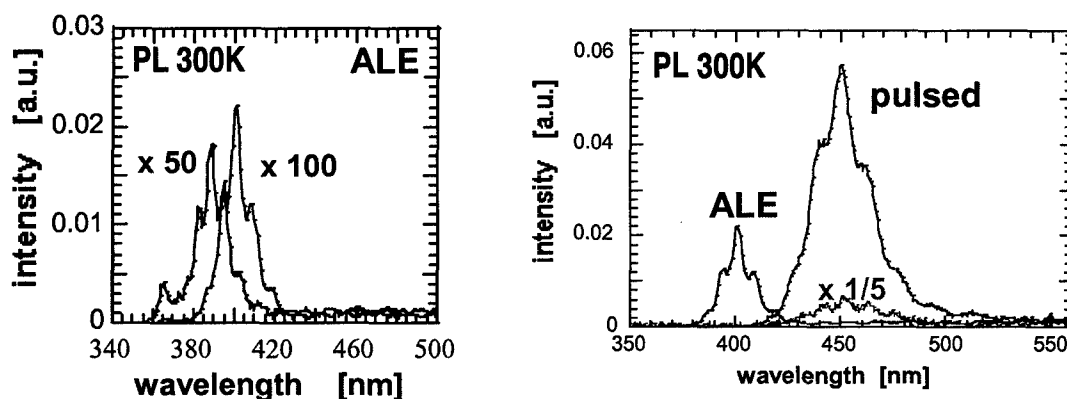


Fig. 8. Photoluminescence spectra of GaN/ InN/ 3nm GaN structures. Left: samples with different InN layer thickness (50 and 100 ALE cycles), right: samples where the InN layer was deposited by ALE (blue), in the pulsed mode (red) and using conventional growth (green).

5. Conclusions

Thin InN and GaN layers could be deposited in a step flow growth mode using pulsed or alternate precursor injection schemes at growth temperatures around 600 °C. Besides the improvements in the structural and morphological quality, the modulated precursor supply also resulted in a reduced incorporation of carbon and oxygen impurities into the layers. However, for device applications the residual impurity concentration in the layer needs to be further reduced. In addition, for the successful deposition of InN containing heterostructures, the lattice mismatch between InN and the adjacent layers has to be lowered to prevent intermixing and strain related degradation of the film quality. Initial experiments showed indeed improvements in the surface morphology when the thin InN layers were deposited on $\text{In}_{0.15}\text{Ga}_{0.85}\text{N}$ (on GaN) layers as illustrated in Fig. 7c.. However, intense research efforts are needed before InN containing heterostructures can be successfully used for device applications.

The results were presented in part in:

S. Keller, I. Ben-Yaacov, S.P. Denbaars, and U.K. Mishra,

Flow modulation epitaxy of InN/GaN heterostructures; towards InN based HEMTs

Proceedings of International Workshop on Nitride Semiconductors, Nagoya, Japan, 24-27 Sept. 2000. Tokyo, Japan: Inst. Pure & Appl. Phys, 2000. p.233-6.

S. Keller, I. Ben-Yaacov, S.P. DenBaars, and U.K. Mishra

Metal-organic chemical vapor deposition of InN

International Symposium on Compound Semiconductors, Monterey, CA, October 2000

References:

- 1) B.P. Keller, S. Keller, D. Kapolnek, W.N. Yiang, Y.-F. Wu, H. Masui, X. Wu, B. Heying, J. S. Speck, U.K. Mishra, and S. P. DenBaars, J. Electron. Mater. **24**, 1707 (1995)
- 2) B. Daudin, F. Widmann, G. Feuillet, Y. Samson, J.L. Roviére and N. Pelekanos, Materials Science Forum, Vols. 264 – 268 (1998) 1177.
- 3) I. Ho and G.B. Stringfellow, Appl. Phys. Lett. **69** (1996) 2701
- 4) R.Y. Korotkov, B.W. Wessels, MRS Fall Meeting. Boston 1999